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NUCLEAR INSTRUMENTS & METHODS IN PHYSICS RESEARCH
Section A

Application of the Renninger effect to X-ray double-plane collimation

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The opportunity of X-ray double-plane collimation due to forbidden Bragg reflex simulated by 3-beam diffraction has been studied, both theoretically and experimentally. It has been shown that the double-plane collimation is provided in both Bragg and Laue cases of forbidden reflex, but in the latter case the degree of the collimation is much higher.

1. Introduction

The majority of modern collimating systems in X-ray optics provide precise angular collimation of X-rays in one plane only (see, e.g., [1–3]). This collimation (let us call it horizontal) is produced by Bragg reflections from single crystals and can be as fine as $\delta\Theta \sim 10^{-1}$–$10^{-2}$ arc sec. At the same time the vertical angular divergence of X-rays is usually confined by slits, pinholes or X-ray mirrors (see review [1]) and has an order no less than 10^2–10^3 arc sec. That suits well for most of the experiments.

However, the progress in X-ray optics has recently been primarily related to non-coplanar cases of X-ray diffraction, such as multiple diffraction [4–7], surface- (grazing incidence) [8–10], back- [11,12] and surface back diffraction [13]. The precise experiments in these geometries require that the incident beam be collimated in both mutually perpendicular planes with an accuracy $\delta\Theta \leq 1''$ ($\leq 10^{-5}$ rad). Moreover, the degree of X-ray monochromatization must be high too: $\delta\lambda/\lambda \leq 10^{-5}$ because the dispersion depending on $\delta\lambda/\lambda$ cannot be compensated in the majority of such experiments.

The above-listed requirements can be met only with an additional Bragg reflection in the second (vertical) plane.

The most straightforward solution of the problem, offered in refs. [7,14], consists in using three Bragg monochromators (Fig. 1a). Crystal 1, mounted horizontally, collimates X-rays in a vertical plane. To prevent the deviation of the beam path from the horizontal plane this crystal must be grooved [15] with an even number of Bragg reflections in the groove. The second crystal, mounted vertically, eliminates the horizontal beam divergence. At last, the third crystal is also placed vertically in the $(+n, +m)$ non-parallel Bragg position with respect to the second crystal. This crystal cuts a narrow interval $\delta\lambda/\lambda$ to eliminate the beam divergence caused by dispersion [#1].

Evidently, the multi-crystal monochromator is rather intricate and requires a careful adjustment. Therefore, it is desirable to obtain the horizontal and

[#1] In ref. [7] the third monochromator was erroneously leaved out.

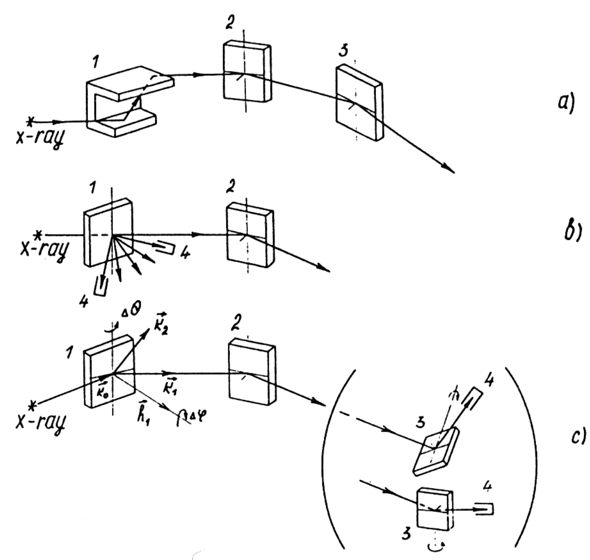

Fig. 1. X-ray double-plane collimation systems: (a): with two-beam reflections from three crystals, (b): with six-beam Borrmann effect, (c): with three-beam simulation of forbidden reflex (Renninger effect). 1,2,3: collimating crystals. In (c): 3,4: the analyzer and detector in our experiment.

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the vertical collimation simultaneously with the help of one crystal by using multiple diffraction. But the problem is that such a multiple diffraction case should be employed when the X-ray beam leaves the crystal in the multiple diffraction point only and does not leave in the other angular domains where the multiple diffraction splits into two-beam components.

As far as we know, only two schemes of multiple diffraction collimation have been proposed.

One of them is based on the six-beam Laue-case diffraction in a thick crystal [5,6]. In view of the fact that the six-beam anomalous transmission effect is many times stronger than the two-beam one, the X-rays leave the crystal only in the angular domain of six-beam Borrmann effect. As a result, a double-plane collimation is produced (see Fig. 1b). The complete device includes also a second crystal mounted vertically in two-beam Bragg position to cut a narrow $\delta\lambda/\lambda$ interval. We appreciate this nice idea but consider that the complexities related to six-beam Borrmann effect implementation (e.g. thermal instability) make its wide practical application uncertain.

Another way of the double-plane collimation suggested in refs. [16-19] is based on a forbidden Bragg reflection simulated by multiple diffraction (the Renninger effect [20]). Taking into account that the forbidden reflex is excited in the multiple diffraction domain only, the respective X-ray beam is collimated in two planes [#2]. The complete collimator is to be equipped with the second vertically mounted two-beam monochromator for the elimination of dispersion (Fig. 1c).

A "forbidden" collimator has been used in ref. [24] for the grazing incidence diffraction studies. In ref. [25] we have carried out a rough measurements of collimation parameters for this type of collimator.

It should be noted that the theoretical studies of the "forbidden" collimator bring some contradictory conclusions. In particular, the calculations of the collimation parameters in two-beam approximation in refs. [17,18] predict a small beam divergence in both planes. On the contrary, the three-wave computations [19] show long tails in the angular distribution. The precision of the experiment [25] is not enough to resolve these contradictions.

[#2] The coplanar case of Renninger effect was analyzed in refs. [21-23]. In this case the incident X-ray beam and all the reciprocal lattice vectors are coplanar and the deviations of X-rays from this plane as large as $\simeq \pm 10'$ have minor effect on the multiple diffraction. Therefore, the precise vertical collimation is not achieved. But, on the other hand, the coplanar case is very sensitive to variations of $\delta\lambda/\lambda$ and produces X-ray monochromatization with an accuracy of $\delta\lambda/\lambda \simeq 10^{-5}$.

Taking into account this uncertainty and considering the Renninger effect to provide the simplest method of X-ray double-plane collimation we carry out a detailed experimental and theoretical study of the effect. This work is also aimed to give some recommendations for applications of the "forbidden" monochromator.

In section 2 the effects of the vertical and the horizontal dispersion in the "forbidden" monochromator are discussed and a way for their elimination is proposed.

In section 3 the experimental procedure is described and the experimental results are presented.

In section 4 we compare the experiment with the computations based on the three-wave dynamical diffraction theory and discuss the ways to optimize the collimation produced by the "forbidden" monochromator.

2. Dispersion analysis

2.1. Procedure of the Renninger effect implementation

To facilitate the following discussion let us briefly overview the Renninger effect.

As known (see, e.g., ref. [27]), the scattering amplitude F_h of X-ray reflection from the crystal planes with a reciprocal vector $\boldsymbol{h}$ is proportional to the following sum over all atoms in a crystal unit cell:

$$F_h \sim \sum_i f_h^{(i)} \exp(-iW_h^{(i)}) \exp(i\boldsymbol{h}\cdot\boldsymbol{r}^{(i)}) . \tag{1}$$

Here $f_h^{(i)}$ and $\boldsymbol{r}^{(i)}$ are the amplitudes of X-ray scattering by the ith atom and the coordinate vector of the atom in the unit cell, respectively and $\exp(-iW_h^{(i)})$ Debye-Waller factor.

Due to crystal symmetry the terms in the sum (1) can cancel each other for some reflex, so as F_h becomes zero or nearly zero. This reflex is known to be called "forbidden" [20].

Following Renninger the forbidden reflex can be simulated with the help of multiple diffraction by the following procedure (see Fig. 1c, Crystal 1). At first, rotating the crystal around a vertical axis we set it under the Bragg angle for the forbidden reflex $\boldsymbol{h}_1$. Then keeping θ fixed and rotating the crystal around $\boldsymbol{h}_1$, one can bring into the diffraction position an additional reflex $\boldsymbol{h}_2$. In this case the X-rays, diffracted by planes with $\boldsymbol{h}_2$, strike the exact Bragg condition for the planes with $\boldsymbol{h}_3 = \boldsymbol{h}_1 - \boldsymbol{h}_2$ and leave the crystal in the direction of $\boldsymbol{K}_1$, the diffracted wave vector being $\boldsymbol{h}_1$. Thereby, the multiple diffraction simulation of the forbidden reflex $\boldsymbol{h}_1$ is realized.

2.2. *Effect of X-ray wavelength variations on multiple diffraction point*

For simulation of $\boldsymbol{h}_1$ reflex the X-ray beam must simultaneously satisfy the Bragg conditions for $\boldsymbol{h}_1$ and $\boldsymbol{h}_2$. Therefore the direction of the incident wave $\boldsymbol{K}_0$ of a given wavelength λ is fixed with respect to both the angles θ and φ and the diffracted beam $\boldsymbol{K}_1$ is collimated in double planes.

However, a non-monochromatic X-ray beam still remains uncollimated because the angular position of the multiwave point varies with the wavelength.

To find the variations of θ and φ with λ we can use the formulae from refs. [21] or [28]. Particularly, the following relation was obtained in ref. [28] from the requirement for the incident wave to satisfy two Bragg conditions for $\boldsymbol{h}_1$ and $\boldsymbol{h}_2$:

$$\cos\varphi = \frac{\boldsymbol{h}_2^2 - \boldsymbol{h}_1 \cdot \boldsymbol{h}_2}{2h_{2\perp}\cos\theta}\lambda, \tag{2}$$

where $h_{2\perp}$ is the projection of $\boldsymbol{h}_2$ on the plane normal to $\boldsymbol{h}_1$, φ is the angle between $\boldsymbol{h}_{2\perp}$ and the projection of $\boldsymbol{K}_0$ on the same plane ($\varphi = 0$ in the coplanar case). Taking into account the conditions:

$$(2/\lambda)\sin\theta = h_1 \qquad \text{(Bragg's law)}, \tag{3}$$

$$\boldsymbol{h}_1 - \boldsymbol{h}_2 = \boldsymbol{h}_3, \tag{4}$$

$$\boldsymbol{h}_{2\perp} = |\boldsymbol{h}_1 \times \boldsymbol{h}_2|/h_1 = |\boldsymbol{h}_2 \times \boldsymbol{h}_3|/h_1, \tag{5}$$

we can rewrite Eq. (2) in the following form:

$$\begin{aligned}\cos\varphi &= -\tan\theta(\boldsymbol{h}_2 \cdot \boldsymbol{h}_3)/|(\boldsymbol{h}_2 \times \boldsymbol{h}_3)| \\ &= -\tan\theta/\tan\theta_{23},\end{aligned} \tag{6}$$

where θ_{23} is the angle between $\boldsymbol{h}_2$ and $\boldsymbol{h}_3$. Applying a simple geometrical analysis one can find that $\theta_{23} = 180° - \theta_{max}$, θ_{max} being the maximum Bragg angle of reflex $\boldsymbol{h}_1$ in the given three-beam configuration (realized in the coplanar case).

Differentiating Eqs. (3) and (6) with respect to $\delta\lambda$, we find the variations of θ and φ with λ:

$$\delta\theta = -(\delta\lambda/\lambda)\tan\theta, \tag{7}$$

$$\delta\varphi = (\delta\lambda/\lambda)\cot\varphi/\cos^2\theta. \tag{8}$$

As defined above, the angle φ determines the vertical deviations of the incident beam projection on the plane normal to $\boldsymbol{h}_1$. It is easy to see that when λ varies the vector $\boldsymbol{K}_0$ itself turns through the angle $\delta\theta_V$ which is less than $\delta\varphi$:

$$\delta\theta_V = (\delta\lambda/\lambda)\cot\varphi/\cos\theta. \tag{9}$$

It is convenient to rewrite Eq. (9) in the following form:

$$\delta\theta_V = -(\delta\lambda/\lambda)\tan\theta_V, \tag{10}$$

where $\theta_V = \arctan(-\cot\varphi/\cos\theta)$ is the effective Bragg angle characterizing the vertical dispersion of the "forbidden" monochromator.

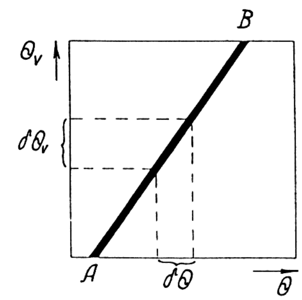

Fig. 2. Elimination of polychromatic beam double-plane dispersion with the secondd crystal. AB: cross section of beam spread after the first crystal, $\delta\theta$: the horizontal spread limited by the second crystal, $\delta\theta_V$: the related vertical spread.

Thus, the variations of λ cause both horizontal and vertical dispersion. At small $\delta\lambda/\lambda$ the ratio of vertical to horizontal dispersion is constant:

$$R_H^V = \delta\theta_V/\delta\theta = -\cot\varphi/\sin\theta. \tag{11}$$

The derived equations show that if non-monochromatic and divergent X-rays are reflected from the "forbidden" monochromator they are converted into a fan of monochromatic rays with linear variation of the wavelength from ray to ray and the slope R_H^V to the horizontal plane. However, if the produced beam undergoes a second Bragg reflection eliminating its non-monochromaticity, then both the horizontal and the vertical beam divergences are avoided (see Fig. 2). For this aim an additional vertically set monochromator in the two-beam dispersive Bragg position can be used.

Using various secondary planes $\boldsymbol{h}_2$, one can vary R_H^V and, thereby, the degree of vertical collimation.

3. Experiment

The experimental study was carried out on an automatized triple-crystal diffractometer equipped with an additional goniometer for precise scans around horizontal axis (see Fig. 1c). The X-ray source was conventional 1.5 kW tube with Cu anode.

Crystal 1 (Ge wafer with (111) surface orientation) was aligned to (222) forbidden Bragg reflex simulated by three-beam diffraction $(222)/(1\bar{1}\bar{1})/(133)$. This multi-beam configuration was selected from the other combinations of secondary reflexes simulating (222) because it provided one of the most intensive peaks (see, e.g., the diagram in ref. [28]). The application of less intensive multi-beam combinations which could probably provide better collimation was restricted by the small intensity of the X-ray tube. The calculated parameters of the used combination were: $\theta = 28.14°$, $\theta_V = 31.37°$, $\varphi = 61.75°$, $R_H^V = 1.14$.

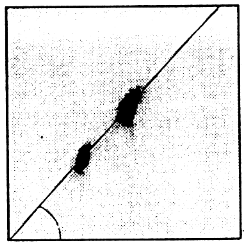

Fig. 3. Measured topograph of beam spread after the "forbidden" monochromator. Straight line indicates the slope of the spread fan.

Crystal 2 was mounted in the dispersive Bragg position with respect to the Crystal 1 to eliminate the dispersion in λ and, thereby, to produce a double-plane collimation. The two-beam (220) reflection from Si wafer cut along (110) was applied. Parameters of the second reflection were: Bragg angle $\theta_2 = 23.65°$, rocking curve halfwidth: $\Omega_2 = 5.1''$.

Finally, Crystal 3 was set either vertically or horizontally to analyze the angular spread of the beam formed by the collimating system. The symmetrical (400) Bragg reflex from Si wafer $\theta_3 = 34.57°$, $\Omega_3 = 3.4''$ was used.

At the beginning of the experiment a topogram of the beam spread after Crystal 1 was measured (Fig. 3). The spots in the figure are the images of the X-ray tube focus produced by Cu K_{α_1} and Cu K_{α_2} characteristic wavelengths of the X-ray spectrum. The straight line drawn through the spots has a slope to the horizontal plane of 1.1 ± 0.1, which correlates quite well with the calculated value.

We should note that there are two equivalent multiwave combinations $(222)/(1\bar{1}\bar{1})/(133)$ and $(222)/(\bar{1}\bar{1}1)/(331)$ characterized by the opposite signs of the vertical dispersion and called IN and OUT [19]. The measured topographs for these combinations give mirror images with respect to horizontal plane in good agreement with the calculations.

The results of the analyzer scans are presented in Fig. 4. The measured angular spreads of the beam according to the figure are: $\delta\theta = 11.5''$, $\delta\theta_V^{IN} = 34.5''$, $\delta\theta_V^{OUT} = 42.5''$. The difference between $\delta\theta_V^{IN}$ and $\delta\theta_V^{OUT}$ is due to the opposite signs of their vertical dispersion and indicates an insufficient monochromaticity of the beam. The matter is that the dispersions of the Crystal 1 and the analyzer crystal are practically compensated (the angles θ_V and θ_3 are close) in the case IN, whereas in the case OUT the dispersions are added and the measured curves are wider.

Thus, our experiment has demonstrated that the proposed system really collimated X-rays in two planes, but the horizontal collimation was much better than the vertical one.

In relation to the obtained data the most important question arose: was the relatively crude vertical collimation attributed to the used multiwave combination or to the Renninger effect in general? Since our experimental potentialities were limited by low intensity of X-ray tube, we tried to answer this question theoretically applying the precise computations based on the three-beam dynamical diffraction theory and computation algorithm described in refs. [26,27].

4. Three-beam computations and discussion

4.1. Simulation of the experiment

At first we carried out three-beam computations for the configuration used in the experiment (see Fig. 5) [#3]. As can be seen in the picture, the used three-beam case really provides much better horizontal collimation than the vertical one. Moreover, the computed topograph in Fig. 5b demonstrates undesirable fine structure in the form of two hyperbolic "wings" with a gap in the centre between them.

To compare the theoretical and experimental data the angular distribution in Fig. 5 was integrated over θ_V (dashed line in Fig. 4a) and over θ (dashed line in Fig. 4b). In this case the fine structure disappeared, but the difference in the collimation values remained: $\delta\theta^{Th} = 3.0''$, $\delta\theta_V^{Th} = 28.6''$.

We should note a considerable difference between the measured and computed horizontal spreads in Fig. 4a. It is likely due to the dispersion and convolution of reflections from Crystals 1 and 2.

Comparing the vertical spreads in Fig. 4b one can see that the experimental curve in the case IN has almost the same halfwidth as the theoretical one, whereas the curve OUT is wider. We attribute this fact to the above mentioned compensation of the vertical dispersion in the former case.

It should also be noted that the values $\delta\theta$ and $\delta\theta_V$, calculated with approximate formulae given in refs. [18,19] on the basis of perturbation theory in two-beam approximation, are considerably underestimated: $\delta\theta^{Chang} = 3.7''$ and $\delta\theta_V^{Chang} = 8.4''$. These values correlate neither with the experiment nor with the exact theory. We attribute this mismatch to the inadequacy of the perturbation theory in the centre of the multi-beam peak.

[#3] The computations assumed monochromatic X-rays and did not account for the dispersion and convolutions of rocking curves.

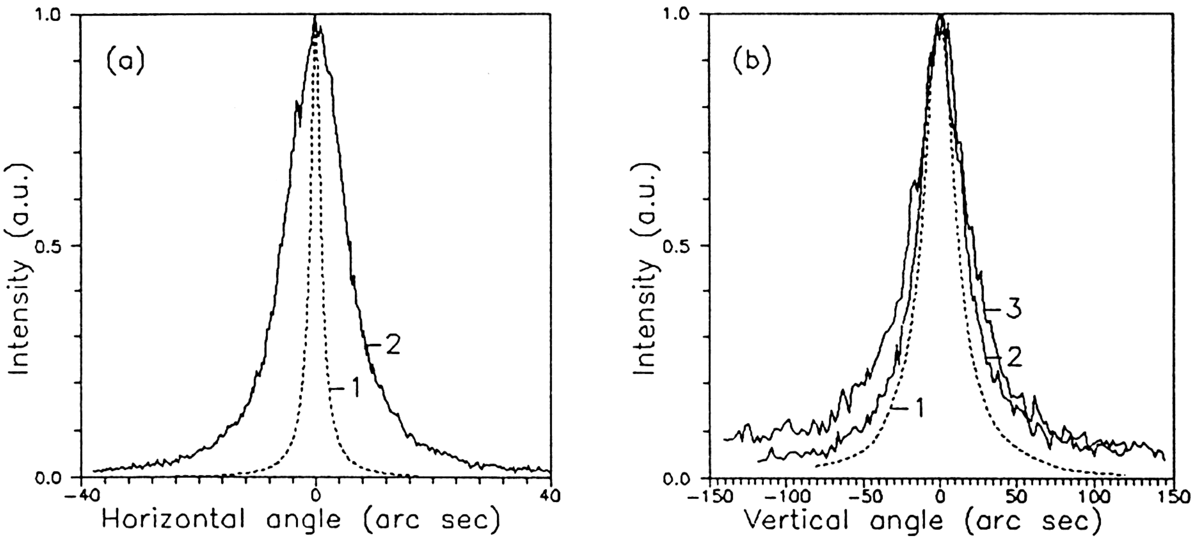

Fig. 4. Experimental curves of (a) horizontal and (b) vertical spreads of the beam produced by double-crystal monochromator. 1: theoretical curve, 2,3: experiment in cases IN and OUT respectively.

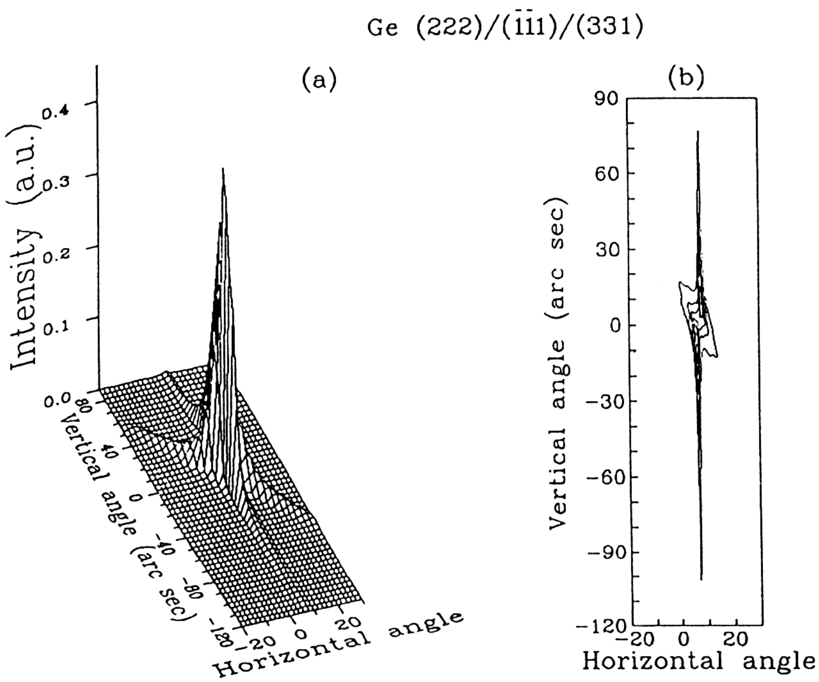

Fig. 5. Computed shape of three-wave peak (222)/($1\bar{1}\bar{1}$)/(133) used in the experiment. (a): spatial view, (b): topograph.

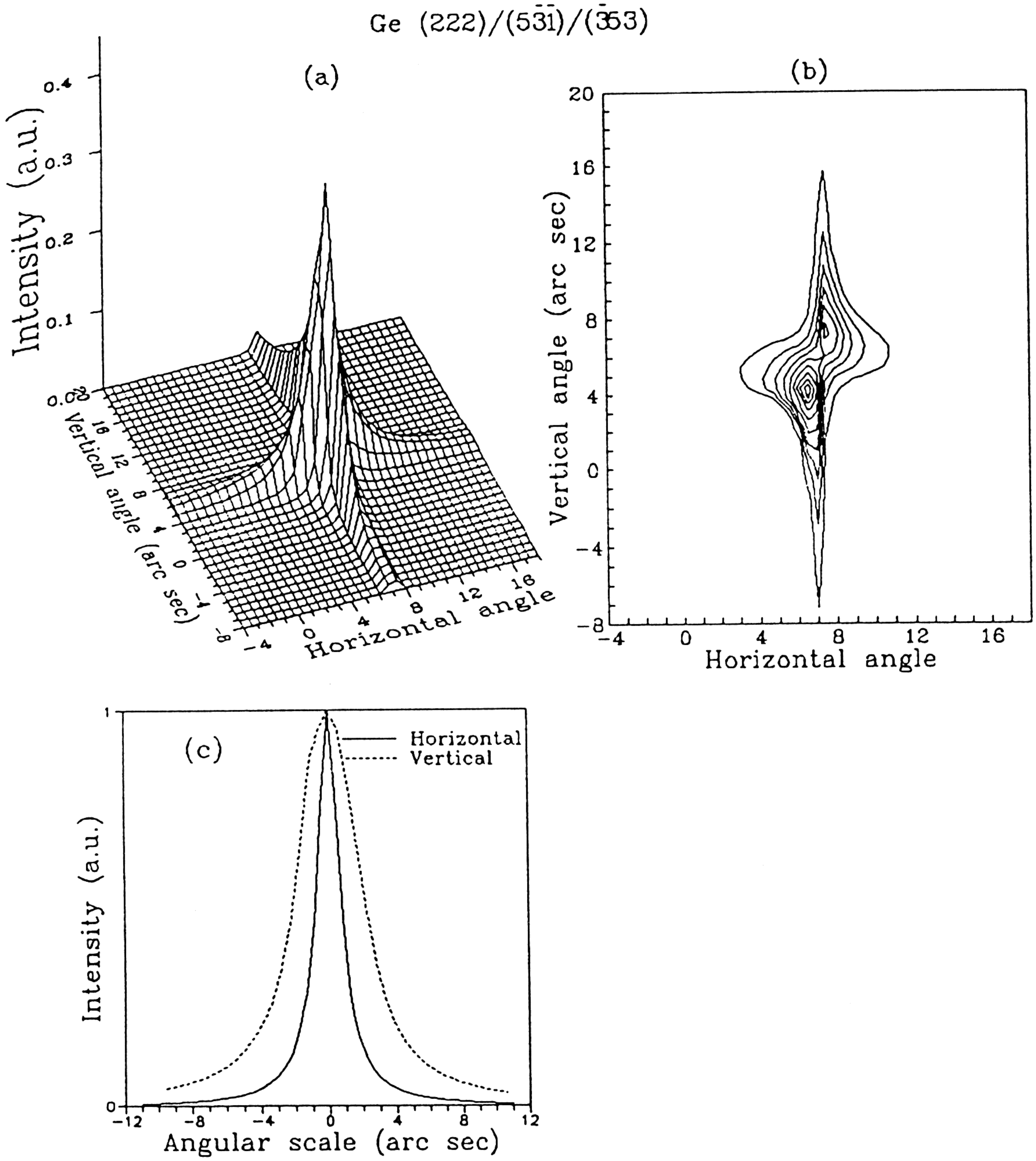

Fig. 6. The same as in Fig. 5 for peak (222)/(53$\bar{1}$)/($\bar{3}$53). (c): horizontal and vertical integrated distributions.

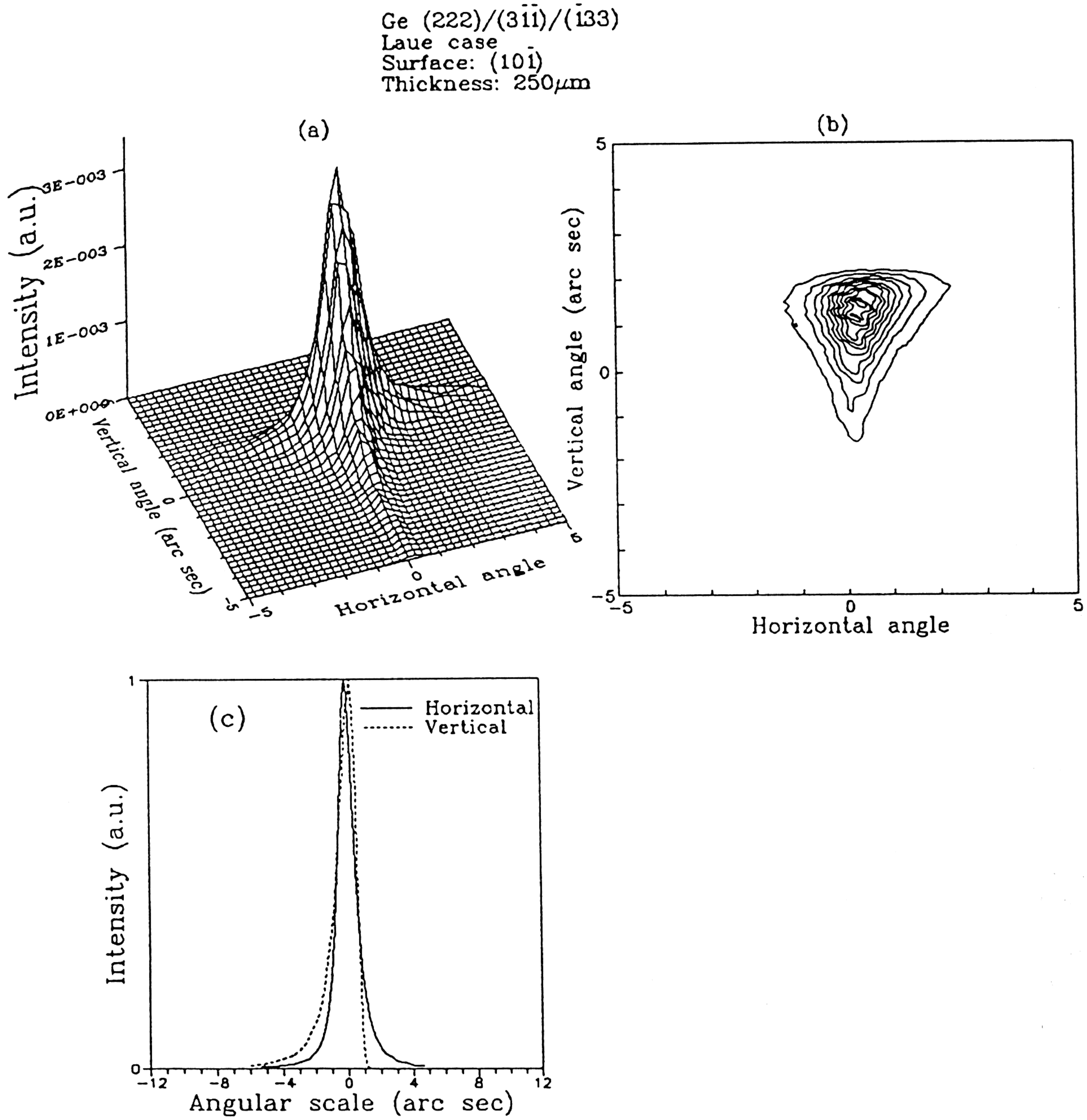

Fig. 7. The same as in Fig. 5,6 for Laue-case peak (222)/(3$\bar{1}\bar{1}$)/($\bar{1}$33).

4.2. Searching for optimal three-beam configuration

To optimize the collimation parameters we carried out the computations for all secondary planes simulating (222) Bragg-case reflex in Ge. The dependence of $\delta\theta_V/\delta\theta$ upon the crystal surface misorientations from (111) was analyzed too. One of the best combinations is shown in Fig. 6.

Summarizing these computations we can make the following conclusions:

(i) Some combinations provide much better $\delta\theta_V/\delta\theta$ than the case used in the experiment, but in any case the condition $\delta\theta_V/\delta\theta > 2$ is kept.

(ii) All the peaks display the two-wings fine structure with long tails along the borders of the wings as reported earlier [26].

(iii) An asymmetrical diffraction geometry can alter $\delta\theta$ and $\delta\theta_V$, but cannot change qualitatively their ratio. This result disproves the statement of ref. [18] based on the perturbation theory. In some cases it causes an extension of the gap between the hyperbolic wings.

Thus, the classical Renninger effect which is the three-beam simulation of the Bragg-case forbidden reflex displays several characteristics impeding its application for the fine X-ray double-plane collimation required in multiple diffraction experiments. Nevertheless, it can be used, for example, in the grazing incidence diffraction studies where somewhat lower vertical collimation is required. Besides, some parasitic effects, such as long tails, can be eliminated if the Renninger effect is realized in a grooved crystal.

4.3. Laue case "forbidden" monochromator

We also analyzed the possibility of X-ray double-plane collimation with the Laue-case forbidden reflex. It turned out that at $\mu t > 8$ the three-beam simulated Laue reflex can provide the excellent collimation with $\delta\theta_V/\delta\theta \sim 1$ without long tails and the central gap (see Fig. 7).

Though the intensity maximum in this case is by 10^1–10^2 times lower than in the Bragg case, this would not be a serious disadvantage because the double-plane collimation experiments, in any case, are feasible with intense synchrotron radiation.

5. Conclusions

The possibility of X-ray double-plane collimation with Renninger effect has been analyzed both theoretically and experimentally.

It has been shown that the most effective collimator is based on three-beam simulation of the Laue-case reflex, i.e. is a combination of collimating systems proposed in refs. [5,6] and refs. [17–19]. Its advantage over the method of refs. [5,6] is in the employment of three-beam diffraction instead of six-beam one. The advantage over refs. [17–19] is in the elimination of undesirable fine structure and long tails.

The contributions of polychromatic dispersion into horizontal and vertical spread have been determined.

The experiment in general confirms the possibility of X-ray double-plane collimation with the Bragg-case Renninger effect. In the Laue case the implementation of the experiment requires a powerful X-ray synchrotron source.

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